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Oxidative Addition Reactions of I_2 with $[HIr_4(CO)_{10-n}(PPh_3)_n(\mu\text{-}PPh_2)]$ (n = 1 and 2) and Crystal and Molecular Structure of $[HIr_4(\mu\text{-}I)_2(CO)_7(PPh_3)(\mu\text{-}PPh_2)]$

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As reações dos compostos $[HIr_4(CO)_{10-n}(PPh_3)_n(\mu\text{-}PPh_2)]$ [n = 0, (**1**); 1, (**2**) e 2, (**3**)] com I_2 foram investigadas. O composto **1** não reage, porém, a substituição de ligante(s) CO por PPh_3 leva à ativação do cluster. De fato, ambos os compostos **2** e **3** reagem com I_2 em condições brandas com a formação de $[HIr_4(\mu\text{-}I)_2(CO)_7(PPh_3)(\mu\text{-}PPh_2)]$ (**4**), como resultado da adição oxidativa de I_2 e dissociação de dois ligantes CO ou de um CO e uma PPh_3, respectivamente. A estrutura molecular de **4**, determinada por um estudo de difração de raios X, exibe um arranjo metálico na forma de uma borboleta, cujas asas se encontram ligadas pelo ligante em ponte μ-PPh_2; os átomos que formam o corpo da borboleta se encontram ligados a um ligante μ-H, e cada uma das duas asas contém um ligante iodo ligado em ponte; todos os átomos metálicos contêm dois ligantes CO terminais, exceção feita de um dos átomos do corpo da borboleta que contém um ligante CO e um PPh_3. O cluster exibe a menor distância média de ligação Ir–Ir observada até hoje em derivados do composto **1**, o que está de acordo com o fato de o estado de oxidação médio dos centros metálicos (+1) ser relativamente alto para clusters metálicos carbonílicos.

The reactions of the cluster compounds $[HIr_4(CO)_{10-n}(PPh_3)_n(\mu\text{-}PPh_2)]$ [n = 0, (**1**); 1, (**2**) and 2, (**3**)] with I_2 have been investigated. Compound **1** does not react, however, the presence of PPh_3 in place of CO ligand(s) activates the cluster. Both compounds **2** and **3** react with I_2 under mild conditions to give $[HIr_4(\mu\text{-}I)_2(CO)_7(PPh_3)(\mu\text{-}PPh_2)]$ (**4**), as the result of oxidative addition of I_2 and dissociation of two CO ligands, or one CO and one PPh_3 ligands, respectively. The molecular structure of **4**, determined by an X-ray diffraction study, exhibits a butterfly arrangement of iridium atoms with the wings spanned by a μ-PPh_2 ligand, the hinge bridged by a μ-H ligand, two hinge to wing tip edges bridged by iodine atoms and all metal atoms bearing two CO ligands, with the exception of one of the hinge atoms that contains a CO and a PPh_3 ligands. This cluster exhibits the shortest average Ir–Ir bond length [2.698(2) Å] observed so far for a derivative of **1** and this is in accord with the relatively high average oxidation state of its metal atoms (+1) for a carbonyl cluster compound.

Keywords: iridium cluster, oxidative addition, iodine, X-ray molecular structure

Introduction

The cluster compound $[HIr_4(CO)_{10}(\mu\text{-}PPh_2)]$ (**1**) undergoes facile CO substitution with phosphines and phosphites to yield the mono- and di-substituted derivatives $[HIr_4(CO)_{10-n}L_n(\mu\text{-}PPh_2)]$ (n =1 and 2).[1] Our recent studies indicate that these derivatives behave differently from **1** in the presence of molecules or fragments that may undergo oxidative addition. For example, although **1** does not react with alkynes and alkenes,[2] phosphines containing unsaturated fragments, such as $Ph_2PC\equiv CPh$, can interact further with **1** to yield μ_4-η^3-Ph_2PCCPh containing species and products resulting from P-C bond activation,[3,4] further hydrometallation[5] or P-C bond formation.[6] Furthermore, we have found that oxidative addition of the $PhP(CPh)_2$ ring is extremely sensitive to the metal frame electronic density and can be delicately tuned by the presence of PPh_3.[7] We report herein our comparative studies of the reactivity of **1** and $[HIr_4(CO)_{10-n}(PPh_3)_n(\mu\text{-}PPh_2)]$ (n =1, **2** and n=2, **3**) with I_2, MeI and O_2, which were carried out in an attempt to make a further parallel between their chemistry and that of Vaska's compounds.[7,8]

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Experimental

Iridium carbonyl (Strem Chemicals), triphenylphosphine and iodine (Aldrich), were used as purchased. THF was dried over sodium and benzophenone, CH_2Cl_2 over CaH_2, hexane and toluene over sodium. All solvents were freshly distilled and degassed before use. The compounds $[HIr_4(CO)_{10}(\mu\text{-}PPh_2)]$ (**1**), $[HIr_4(CO)_9(PPh_3)(\mu\text{-}PPh_2)]$ (**2**) and $[HIr_4(CO)_8(PPh_3)_2(\mu\text{-}PPh_2)]$ (**3**) were prepared by published methods.[9] The progress of the reactions was monitored by analytical TLC (pre-coated plates, silica gel F254, 0.25 mm thick; E. Merck) and IR spectroscopy. The separation and purification of the reaction products were carried out by preparative TLC (2mm thick glass plates 20X20 cm, silica gel GF 254; Fluka). The reactions and manipulations were performed in typical Schlenk systems, under inert atmosphere of argon. Infrared spectra were recorded on a Bomem (FT-IR Michelson) spectrophotometer between 2200 and 1600 cm^{-1} (ν_{CO}), $^{31}P\{^1H\}$ and ^1H spectra on a Bruker AC 300P spectrometer using, as references, 85% H_3PO_4 (external) for the former and $SiMe_4$ for ^1H.

*Reaction of **2** with I_2: preparation of $[HIr_4(\mu\text{-}I)_2(CO)_7(PPh_3)(\mu\text{-}PPh_2)]$ (**4**)*

A solution of I_2 (7 mg, 0.027 mmol) in toluene (20 mL) was slowly added to a solution of **2** (40 mg, 0.027 mmol) in the same solvent (20 mL), at 25° C, and the resulting mixture was kept under stirring for about 3h at room temperature. The solvent was then evaporated under vacuum and the residue dissolved in CH_2Cl_2 for purification by tlc (CH_2Cl_2/hexane; 2:3), to give some starting material **2** (6 mg, 15%), some brown decomposition material on the plates' base line and compound **4** (22.5 mg, 50%). Compound **4** was recrystallised from CH_2Cl_2/hexane to give red microcrystals of **4** (found: C, 26.6; H, 1.7. Calc. for $C_{37}H_{26}O_7P_2Ir_4$: C, 26.9; H, 1.6%); IR ν_{CO}/cm^{-1} 2027s, 2045vs, 2012s, 2001s (hexane); ^1H NMR ($CDCl_3$, 300 MHz) δ -15.5 (dd, *J*(PH) 8 and 2 Hz, μ-H), 6.4-8.0 (m, Ph); $^{31}P\{^1H\}$ NMR ($CDCl_3$,121.4 MHz) δ 11.1 (d, *J*(PP) 8, PPh_3), 62.1 (d, μ-PPh_2).

*Reaction of **3** with I_2*

The reaction was carried out following same procedure described above, using equimolar toluene solutions of I_2 (8 mg, 0.031 mmol in 20 mL) and cluster **3** (50 mg, 0.031 mmol in 20 mL). The mixture was kept under stirring at the same temperature, but for 5 h. Separation of the crude mixture was carried out as described above and yielded the starting material **3** (7.5 mg, 15%) and cluster **4** (20.7 mg, 40%); a number of very low yield products were separated out on the tlc plates, but were not isolated.

*Attempts at reacting **2** and **3** with O_2*

Oxygen was bubbled through stirred solutions of **2** (40 mg, 0.027 mmol) and **3** (50 mg, 0.031 mmol), in toluene (20 mL) at 45 °C. After 24 h no changes were detected in the colour or in the $^{31}P\{^1H\}$ NMR spectra of the solutions.

*Attempts at reacting **2** and **3** with MeI*

A large excess of MeI (~30 equiv) was added to solutions of compounds **2** (40 mg, 0.027mmol) and **3** (50 mg, 0.031 mmol) in toluene (20 mL) and the mixtures were kept at 20 °C. After 24 h no changes were detected in the colour or in the $^{31}P\{^1H\}$ NMR spectra of the solutions. The temperature of both solutions was then gradually increased to 70 °C, but no reaction was observed after 3 days.

Crystal structure determination

Crystal data and details of measurements for compound **4** are summarised in Table 1. Diffraction intensities were collected at room temperature on an Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator (MoKα, λ = 0.71069 Å). Intensity data were reduced to F_0^2. Crystals were obtained from CH_2Cl_2/hexane by slow evaporation. The cluster was found to cocrystallise with one water molecule per formula unit. The structure was

Table 1. Crystallographic data for compound **4**

	4
Formula	$C_{37}H_{28}I_2Ir_4O_8P_2$
M	1685.13
T/K	293(2)K
Crystal system	Triclinic
Space group	P-1
a/Å	11.088(4)
b/Å	11.912(9)
c/Å	18.096(9)
α/°	72.13(5)
β/°	74.47(4)
γ/°	66.36(5)
U/Å^3	2055(2)
Z	2
D_c/g cm^{-3}	2.723
μ/mm^{-1}	14.538
Independent reflections	5596
Absorption correction	Ψ-scan on 8 reflections χ>80°
*wR*2 (all data)	0.1756
*R*1[*I* >2σ(*I*)]	0.0573

solved by direct methods, followed by least-squares refinements. The crystallographic program SHELXL-97[10] was used for all calculations. All phenyl rings were refined as rigid groups. The hydrogen atoms were added in calculated positions (C_{sp^2}-H 0.93 Å) and refined 'riding' on the corresponding C atoms. For graphical representations the program SCHAKAL99[11] was used.

Results and Discussion

*Reactions of [$HIr_4(CO)_{10}(\mu$-$PPh_2)$] (**1**), [$HIr_4(CO)_9(PPh_3)$-(μ-PPh_2)] (**2**) and [$HIr_4(CO)_8(PPh_3)_2(\mu$-$PPh_2)$] (**3**) with I_2, MeI and O_2*

Treatment of compounds **2** and **3** with equimolar amounts of iodine, in toluene, at 25 °C gave in both cases the air stable red compound [$HIr_4(\mu$-$I)_2(CO)_7(PPh_3)(\mu$-$PPh_2)$] (**4**) in 50 and 40% yields, respectively, after tlc and crystallization from CH_2Cl_2/hexane. In both cases the starting materials were recovered (~ 15%), but the yields of the reactions could not be improved by using excess I_2 which led to an increase in the number of side and decomposition products visualized on the tlc plates.

Under the same conditions, compound **1** did not react with I_2 and under more forcing conditions, decomposition of **1** was observed. Thus, once again,[7] activation of cluster **1** towards oxidative addition was achieved upon CO substitution with PPh_3. However, this process cannot be generalized: indeed, compounds **1**-**3** did not react with MeI under a variety of conditions (25-70 °C). Furthermore, contrarily to our expectations, neither compound **2** nor cluster **3** reacted with O_2 in toluene, even at 45 °C, for 24h.

Compound **4** was first characterized by a combination of IR and ^1H and ^{31}P NMR spectroscopy and satisfactory microanalysis (see Experimental). Only terminal ν_{CO} bands are observed in the IR spectrum. The $^{31}P\{^1H\}$ NMR spectrum exhibits two doublets at δ 62.1 [*J*(PP) 8Hz] and 11.1 attributed to the μ-PPh_2 and PPh_3 phosphorus nuclei, respectively. The shift of the μ-PPh_2 phosphorus resonance to lower frequency compared to those of both starting materials (> δ 250) suggested substantial increase in the μ-PPh_2 bridged Ir–Ir distance.[12] The ^1H NMR spectrum shows a resonance due to a hydride ligand at δ -15.5 [*J*(HP) 8 and 2 Hz] and a multiplet at δ -7.0-8.0 due to the phenyl hydrogens. The small coupling observed between the hydride and the μ-PPh_2 phosphorus nucleus compared with those of the starting materials [*J*(HP) > 50Hz] indicated that in **4** the two ligands are not oriented on the same plane as in clusters **1**-**3**.[1] Because the spectroscopic data did not define the structure of **4**, a single-crystal X-ray diffraction study was undertaken.

*Crystal and molecular structure of **4***

The molecular structure of **4** in the solid state is shown in Figure 1, together with the atomic labelling scheme. Relevant structural parameters are shown in Table 2. Cluster **4** exhibits a butterfly arrangement of metal atoms [dihedral angle 82.4(5)°] with the wings spanned by a PPh_2 bridging ligand, as previously observed for [$Ir_4(CO)_8(PCy_3)(\mu$-$Ph_2PC(H)CPh)(\mu$-$PPh_2)$] (**I**)[4] and [$Ir_4(CO)_9(\mu$-$PhPCPh{=}CHPh)(\mu$-$PPh_2)$] (**II**).[7] The bridging PPh_2 ligand is symmetrically bonded [Ir(3)–P(2) 2.269(5) and Ir(4)–P(2) 2.274Å] and the non-bonding Ir(3)**…**Ir(4) distance, 3.528(2)Å, is shorter than in **I** and **II** [3.753(1) and 3.598(4) Å, respectively]. The Ir–Ir bond lengths range

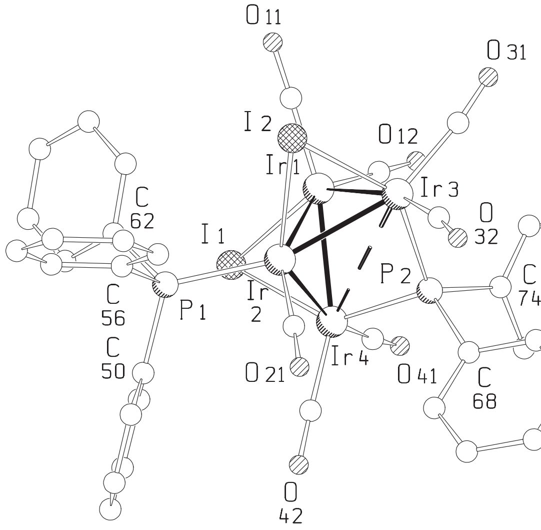


Figure 1. Molecular structure of [$HIr_4(\mu$-$I)_2(CO)_7(PPh_3)(\mu$-$PPh_2)$] (**4**).

Table 2. Selected intramolecular distances (Å) and angles (°) for [$HIr_4(\mu$-$I)_2(CO)_7(PPh_3)(\mu$-$PPh_2)$] (**4**)

Ir(1)-Ir(2)	2.796(2)	Ir(1)-I(1)	2.735(2)
Ir(1)-Ir(3)	2.711(2)	Ir(4)-I(1)	2.787(2)
Ir(1)-Ir(4)	2.631(2)	Ir(2)-I(2)	2.762(2)
Ir(2)-Ir(3)	2.649(2)	Ir(3)-I(2)	2.753(3)
Ir(2)-Ir(4)	2.715(2)	P(1)-C(62)	1.806(9)
Ir(3)**…**Ir(4)	3.528(2)	P(1)-C(56)	1.814(9)
Ir(2)-P(1)	2.304(5)	P(1)-C(50)	1.823(8)
Ir(3)-P(2)	2.269(5)	P(2)-C(74)	1.81(1)
Ir(4)-P(2)	2.274(5)	P(2)-C(68)	1.822(9)
Ir(3)-P(2)-Ir(4)	101.8(2)	Ir(1)-C(11)-O(11)	178(3)
Ir(1)-Ir(2)-P(1)	111.9(1)	Ir(1)-C(12)-O(12)	176(3)
Ir(3)-Ir(2)-Ir(4)	82.2(1)	Ir(2)-C(21)-O(21)	177(2)
Ir(3)-Ir(1)-Ir(4)	82.62(5)	Ir(3)-C(31)-O(31)	173(2)
Ir(1)-I(1)-Ir(4)	56.91(5)	Ir(3)-C(32)-O(32)	175(2)
Ir(1)-I(2)-Ir(2)	57.41(6)	Ir(4)-C(41)-O(41)	176(2)
		Ir(4)-C(42)-O(42)	177(2)

from 2.631(2) to 2.796(2) Å, the longest bond corresponding to the hinge of the butterfly, [Ir(1)-Ir(2)], where the bridging hydride ligand was located on the basis of potential energy calculations; this distance is only slightly longer than the equivalent μ-H bridged Ir–Ir bonds in **1** and **2** [2.769(3) and 2.785(2)Å, respectively]. The two shortest bonds in **4** correspond to the hinge to wing tip edges bridged by iodine atoms [Ir(1)–Ir(4) 2.631(2) and Ir(2)–Ir(3) 2.649(2)Å], which is rather unexpected, since bridging iodine ligands are invariably associated with elongation of M–M bond lengths.[13,14,15] The two iodine ligands are slightly asymmetric [Ir(1)–I(1) 2.735(2), Ir(4)-I(1) 2.787(2)Å and Ir(2)–I(2) 2.762(2) and Ir(3)–I(2) 2.753(3)Å] and practically perpendicular to the triangular wings of the butterfly [Ir(2)–Ir(1)-I(1) 90.51(6) and Ir(2)–Ir(4)–I(1) 91.12° and Ir(1)–Ir(2)–I(2) 87.24(6) and Ir(1)–Ir(3)–I(2) 89.12(7)°]; the slightly different bond distances seen in the two cases can be correlated to the presence of a PPh_3 ligand on one of the hinge metal atoms [Ir(2)] in place of a CO ligand on the other [Ir(1)]. All seven CO ligands are essentially linear and distributed two on each iridium atom, except for Ir(2) that contains a CO and a PPh_3 ligands. Considering that the two μ-I and the μ-PPh_2 ligands formally donate 3 electrons each to the cluster frame, the PPh_3 and CO ligands contribute with two electrons each and the hydride ligand with one, the cluster as a whole is a 62 electron system which is consistent with the butterfly structure.[16]

The molecules are arranged in the crystal in sort of "chains", so as to direct both iodine ligands towards iodines on adjacent clusters, as show in Figure 2 [I(1)...I(1) 3.925(2), I(2)...(I2) 4.009(2) Å]. Pairs of water molecules are trapped in cavities formed by four cluster units, and interact *via* hydrogen bonds within the pair [O(5) ...O(5) 2.989(5) Å].

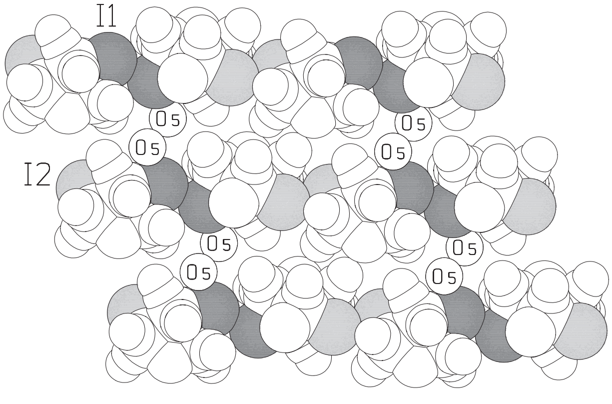
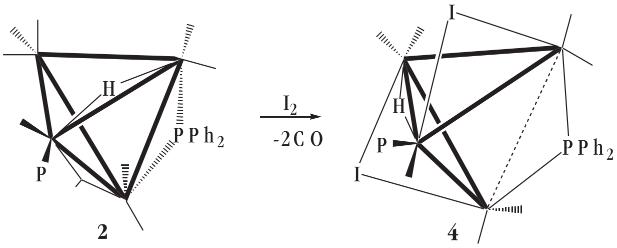


Figure 2. Space-filling representation of the packing arrangement of crystalline **4** (projection in the *ac*-plane), showing the iodine...iodine contacts and the positions of the water molecular pairs in cavities formed by four cluster units. Phenyl groups are not shown for the sake of clarity.

The molecular structure of **4** reveals therefore that addition of I_2 to compounds **2** and **3** was accompanied by cleavage of an Ir–Ir bond and by dissociation of two CO ligands in the case of precursor **2** (see Scheme 1) and one CO and a PPh_3 in that of precursor **3**, *i.e.*, that two ligands (4 electrons) and one Ir–Ir bond (2 electrons) were replaced by two iodine atoms (6 electrons). To our knowledge, formation of **4** is the only example in the literature involving the alternative possible path to M–M bond cleavage, upon addition of I_2, *i.e.* CO dissociation.



Scheme 1.

It is known that the outcome of the reactions of cluster compounds with I_2 depends on the type of cluster and charge distribution within it,[17] but systematic studies are rare. It has recently been shown that the reaction of $[Os_3(CO)_{12}]$ with I_2 proceeds *via* a cationic product, $[Os_3(CO)_{12}(\mu\text{-}I)]^+$, with the μ-I^+ (2 electron donor) in place of a M-M bond, which reacts further to yield the linear species *cis,cis*-$[Os_3(CO)_{12}I_2]$ containing two terminal I atoms (1 e^-_0 donors).[18] Oxidative addition of I_2 to neutral clusters may also result in compounds containing one I atom bonded in a terminal (1 e^-_0 donor) and another, in a bridging μ_2- (3 e^-_0 donor) fashions, and occurs with concomitant cleavage of two M-M bonds.[14,15] Furthermore, excess I_2 has been found to lead to cluster breakdown.[19]

Thus, the CO dissociation path followed in the formation of **4** (instead of the normal M–M cleavage) seems to be at least in part responsible for the shortening of Ir–Ir bonds after addition of I_2 and bridging by the μ-I atoms. We also argue that the short average Ir–Ir bond distance in **4** [2.698(2) Å, excluding the non-bonding distance], the shortest so far observed for derivatives of **1**, is associated to the relatively high formal average oxidative state (that is for carbonyl cluster compounds) of the Ir atoms, +1. In comparison, the Ir atoms in **1** and **2** are on average +1/2 and the average Ir–Ir bond distances in these clusters are much longer, 2.737(2) and 2.751(2) Å, respectively; the average Ir–Ir bond slight expansion upon substitution of the π- acceptor CO in **1** with the σ-donor PPh_3 in **2** is associated with the resulting increase in the cluster electron density. Furthermore, those differences do not seem to be related to the different metal polyhedra in **1** and **2** on the one hand and **4**, on the other, as the average Ir–Ir bond distance in the 62 electron butterflies **I** and **II** [2.749(1) and 2.7914(4) Å, respectively], whose Ir atoms are on average +1/2, are comparable to those observed for **1** and **2**.

Supplementary Information

Crystallographic data (excluding structure factors) for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no CCDC 178269. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44 1223 336033 or e-mail: deposit@ccdc.cam.ac.uk).

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